

1.031.377

## PATENT SPECIFICATION

DRAWINGS ATTACHED

1.031.377



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## COMPLETE SPECIFICATION

## Improvements in or relating to Ceramic Powders

We, THE PLESSEY COMPANY LIMITED, a British Company of 56 Vicarage Lane, Ilford, Essex, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to ceramic powders and particularly to the densification of such powders to facilitate the production of high strength, cold pressed compacts.

Many pure oxide powders consist either of porous particles or particles with irregular shapes and it is difficult to cold press these to give compacts of sufficient green unfired strength to be handled. This problem can be overcome by the use of binders but these may introduce impurities and also cause the final sintered densities to be lowered.

It is an object of the present invention to provide a process for the treatment of pure oxide ceramic powders which increases the tap density of the powder and leads to more efficient packing under pressure. It is a further object of the invention to provide a ceramic powder which may be cold pressed without the use of binders to give compacts of sufficiently high green strength to withstand subsequent handling.

According to the invention, a pure oxide powder is impregnated with a solution of a salt which on heating will decompose to the same material as the powder, the powder then being removed from the solution, dried and subsequently fired to decompose traces of the salt remaining on individual particles thereby depositing further oxide on the original particles such that their bulk density will be increased.

Preferably the mixture of pure oxide powder and solution together forming a ceramic slip is allowed to stand for a sufficient time for the solution to displace air and diffuse into the interior of the porous ceramic par-

ticles. The powder particles are then separated from excess solution and heated to decompose the salt within the particles. The original particles of ceramic powder thus become impregnated with fresh ceramic. Generally, to attain for example a 100% increase in powder density, three or four such impregnations are necessary. The increase in density of the treated ceramic can be estimated by measuring the tap density of the powder. This is obtained by reading the volume of powder tapped, under standard conditions, into a graduated measuring cylinder. The effect of this densification and its usefulness is made evident by examining the pressing characteristics of ceramic powder before and after impregnation.

For a better understanding of the invention reference will be made to the drawing accompanying the Provisional Specification in which;

Figure 1 is a section to a greatly enlarged scale of a particle of ceramic powder before treatment,

Figure 2 is the same section of the particle shown in Figure 1 after impregnation with the solution of decomposable salt, and

Figure 3 is the same section shown after the heating of the impregnated particle.

The ceramic particle shown at 1 has an irregular external shape and includes pores 2. Upon impregnation with a solution of a suitable decomposable salt, the solution lines the walls of the pores and is also retained in external concavities 3 occurring on the surface of the particle. The impregnated ceramic material is then heat treated. During the drying and firing stages, the solvent evaporates and the deposited salt decomposes to produce an oxide layer 4 which lines the pores and concavities of the particle. By repetition of the impregnation, drying and firing stages the refractory layer can be built up until the pores in the particle become substantially filled and the surface irregu-

larities on the particle are largely smoothed out.

A particular application of the process of the present invention will be described with reference to the formation by cold pressing of bodies of alumina powder. Great difficulty has been experienced in the cold pressing, without the use of binders, of bodies of pure alpha alumina in order to obtain compacts of sufficiently high green strength to withstand subsequent handling. This problem is overcome by the following treatment:

100 gm. of pure alpha alumina is made into a slip by the addition of 150 ml. of a solution of aluminium chloride having a specific gravity of 1.2808 (corresponding to 435.5 gm/l. of the anhydrous salt). This slip is allowed to stand for one hour to enable the solution to diffuse into the porous alumina

particles and is then fired at 400°C. to decompose the chloride to gamma alumina. It was found necessary to repeat the process four times so as to yield 100% weight increase of alumina. The powder at this stage is a mixture of the alpha and gamma phases.

To further improve the density and flow properties, the treated powder is milled with distilled water in a rubber lined ball mill with pure alumina balls and then spray dried.

Measurements of tap density on pure alumina, before and after impregnation (0.5-0.6 gm/cc. and 1.1-1.2 gm/cc. respectively) indicate that considerable densification has taken place.

Table 1 shows the variation of packed bulk density and cold pressed density with the amount of alumina added during impregnation.

TABLE I

Weight % of Alumina added during impregnation	Tap Density gm/cc.	Pressed Density (10 t.s.i.) gm/cc.
00	0.58	*1.40
23	0.68	*1.43
44	0.73	1.45
60	0.85	1.52
80	0.92	1.55
100	1.05	1.70
150	1.12	1.83

\*It was difficult to estimate the density of these compacts since they were extremely fragile.

By pressing the impregnated alumina powder at 30 t.s.i., compacts possessing green densities of 2.2 gm/cc. can be achieved. The strength of such compacts is sufficient high to enable them to be machined by turning on a lathe. Thus cold compaction and subsequent machining can be used as a method of producing complicated shapes in the green state.

#### WHAT WE CLAIM IS:

1. A process for the densification of a pure oxide powder in which the powder is impregnated with a solution of a salt which on heating will decompose to the same material as the powder, the powder then being removed from the solution, dried and subsequently fired to decompose traces of the salt

remaining on individual particles thereby depositing further oxide on the original particles such that their bulk density will be increased.

2. A process as claimed in Claim 1 in which the impregnating, drying and firing stages are repeated one or more times.

3. A process as claimed in Claim 1 or Claim 2 for the densification of alumina powder.

4. A process as claimed in Claim 3 in which the alumina powder is impregnated with a solution of aluminium chloride.

5. A process for the densification of a pure oxide powder substantially as hereinbefore described.

6. A pure oxide powder when prepared by a process substantially as hereinbefore des-

cribed.

7. A ceramic compact when prepared from a pure oxide powder substantially as hereinbefore described.

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For the Applicants.

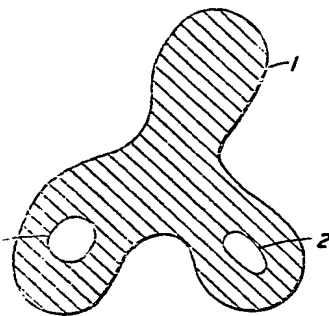
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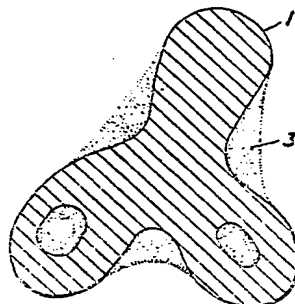
PROVISIONAL SPECIFICATION

1 SHEET

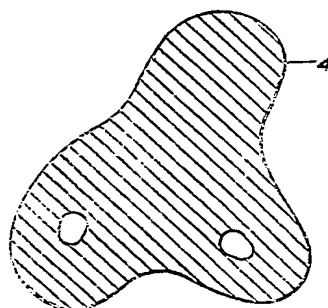
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*FIG. 1.*



*FIG. 2.*



*FIG. 3.*